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Redox-Switchable Phase Tags – Facile Mitsunobu Reactions using Ferrocenyl-Tagged Triphenylphosphine

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Abstract: The use of redox-switched phase tags in ferrocenyl-substituted triphenylphosphine combined with DBAD (di-*tert*-butyl azodicarboxylate) allows high yield (>90%) Mitsunobu transformations without the need for the chromatographic purification of the products. The redox-switchable phosphine can be easily synthesized in two steps from 4-bromoaniline, ferrocene and chlorodiphenylphosphine. It is separated from the reaction mixture by oxidation with iron(III) chloride and can be recycled efficiently by reductive treatment.

Keywords: ferrocene; Mitsunobu reaction; phosphanes; redox reaction; redox-switchable phase tag

Introduction

The implementation of efficient purification strategies in chemical synthesis is now considered to be an important issue for the applicability of chemical transformations. In this respect an enlightening contribution from Curran, reminded chemists that even in the presence of powerful (though time-consuming) separation techniques such as chromatography, both synthesis and separation determine the practical value of a reaction.[1]

However, there are a number of chemical transformations that are highly useful from a synthetic point of view, but are often plagued by purification problems. Infamous in this respect are reactions in which triphenylphosphine is used as a stoichiometric reagent[2] such as the Mitsunobu[3] and the Staudinger reactions[4] or the reduction of primary ozonides.[5]

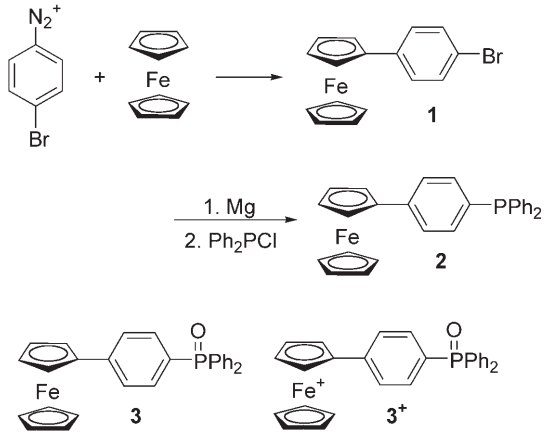
Nonetheless, the Mitsunobu reaction is a powerful synthetic tool for the condensation of an acidic pronucleophile (RXH) and an alcohol (R′OH), due to its wide applicability, stereospecificity and mild reaction conditions. Several strategies have been developed to deal with purification problems in the Mitsunobu reaction such as polymer-supported reagents,[6] basic phosphines,[7] tagged phosphines, various azodicarboxylate reagents,[8,9] fluorous reagents,[10] or phase switching approaches,[11] as described in detail in a recent review by Dandapani and Curran, dealing exclusively with Mitsunobu purification strategies.[12] Ideally such approaches yield pure products obviating chromatography, which is especially important for combinatorial library synthesis.

For the separation of organometallic catalyst complexes we have recently introduced redox-switchable phase tags, which are composed of ferrocenyl groups whose oxidation state can be changed reversibly.[13] In the reduced state such phase tags are neutral and as such are lipophilic units with a good solubility in non-polar solvents. However, on oxidizing the ferrocenyl groups with a mild oxidation reagent, the neutral phase tag is converted into a cationic group, which immediately precipitates from non-polar solvents. A significant advantage in the separation of reagents/catalysts with attached redox-active phase tags is the orthogonality of the solubility determining reductive or oxidative transformation of the phase tag. Furthermore, the redox potential of such phase tags can be adjusted easily to match different reaction conditions and substrates.

We now describe the synthesis and the redox-switched separation of ferrocenyl-tagged triphenylphosphine which is used as a stoichiometric reagent in the Mitsunobu reaction.

Results and Discussion

To be attractive for the user, the synthesis of a redox-switchable triphenylphosphine should be as simple as possible. We have thus devised a two-step procedure (Scheme 1) starting from relatively inexpensive,



Scheme 1. Synthesis of ferrocenyl-tagged PPh_3 (**2**).

commercially available materials such as ferrocene, 4-bromoaniline and diphenylchlorophosphine.

Following the diazotation of 4-bromoaniline, the reaction of the diazonium salt with ferrocene was performed under phase-transfer conditions,[14] to easily yield deca-gram amounts of 4-bromophenylferrocene (**1**) in 74% yield. Next, the Grignard reagent generated from **1** is reacted with diphenylchlorophosphine to result in the formation of the ferrocenyl-tagged triphenylphosphine (**2**) in 83% yield.

The redox potentials of the ferrocenes such as **1** ($E_{1/2}=0.491$ V; $\Delta E=78$ mV), **2** ($E_{1/2}=0.477$ V; $\Delta E=82$ mV) and **3** ($E_{1/2}=0.517$ V; $\Delta E=74$ mV) were determined by cyclic voltammetry (Figure 1).

The choice of the oxidation reagent used to switch the phase tag is critical for the usefulness of the redox-switchable phase tag approach described here. In principle, numerous oxidants are able to convert ferrocene into the respective ferrocene cation, however, the strength of the oxidizer needs to be adjusted to enable quantitative oxidation and to avoid over-oxidation, i.e., decomposition of the ferrocene cation.[15]

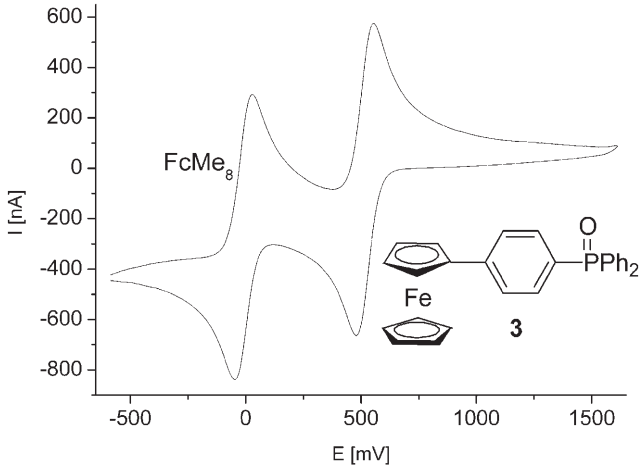


Figure 1. Cyclic voltammogram of **3**, referenced *vs.* $FcMe_8$.

Furthermore, when switching stoichiometric reagents such as **3**, a cheap, easily available and non-toxic oxidant would significantly enhance the impact of this approach. This oxidant should be soluble in the organic solvent used for the Mitsunobu reaction, to effect rapid oxidation of the phase tag.

With these limitations in mind, we decided to use anhydrous iron(III) chloride, which is cheap, readily available and soluble in THF. Most importantly, its oxidizing power is sufficient to quantitatively convert **2** and **3** into the corresponding ferrocenium salts. After the Mitsunobu reaction, solid iron(III) chloride or a solution in THF was added to the reaction mixture in the same solvent to oxidize the phase tag. The various iron salts can now be extracted with water. In this manner > 98% of the ferrocenyl-tagged phosphine oxide $\mathbf{3}^+$ are removed. When using di-*tert*-butyl azodicarboxylate (DBAD) 4 M HCl is next added to destroy the spent Mitsunobu reagent. This highly acidic approach may impose limits with respect to certain acid-labile substrates. However, the redox-switched separation described here is also compatible with other removal strategies which have been applied for modified DEAD (diethyl azodicarboxylate) or DIAD (diisopropyl azodicarboxylate).[12] It is also possible to attach redox-switchable phase tags to the azodicarboxylate by generating the respective ester of ferrocenylmethanol. However, this could lead to complications in the recovery of reagent **2**.

Following the Mitsunobu procedure listed in detail in the Experimental Section the respective products can be isolated in excellent yields (Table 1), with a high purity of the crude products not requiring chromatographic purification.

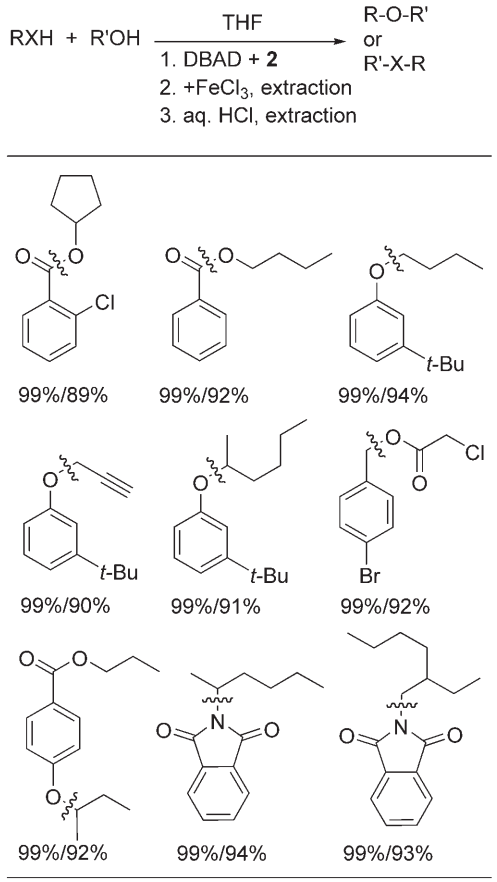
Furthermore, it is very easy to recover $\mathbf{3}^+$ from the aqueous phase by simply adding the reductant sodium thiosulfate which converts $\mathbf{3}^+$ to **3** (Scheme 2). Following the addition of THF and diethyl ether, ferrocene **3** can be re-extracted into the organic phase, from which it is isolated after evaporation. Reduction ($R_3PO \rightarrow R_3P$) of residual **3** with trichlorosilane allows the re-isolation of more than 80% of the initially added ferrocenylphosphine **2**.

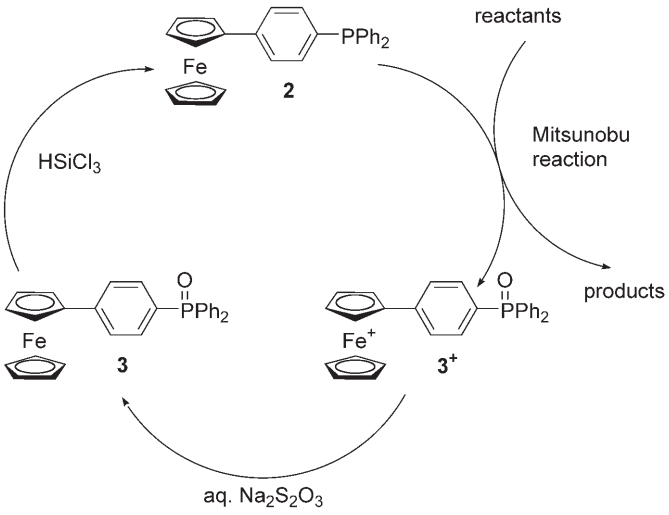
Finally, we were also interested whether the ferrocenyl-group has a significant influence on the Mitsunobu reactivity. Therefore we have monitored the conversion-time curve for the Mitsunobu reactions of chloroacetic acid and 2-propanol using the two different phosphines triphenylphosphine and **2**, which display roughly identical rates of product formation (Figure 2).

Conclusions

In conclusion, we have demonstrated that the use of redox-switchable phase tags allows us to reversibly

Table 1. General scheme of the Mitsunobu reaction and a list of the products synthesized (conversion/isolated yield), wavy lines denote the bonds formed.





Scheme 2. Recycling of ferrocenyl-tagged PPh_3.

modify the solubility properties of a substituted triphenylphosphine enabling the facile separation of the corresponding phosphine oxide. In combination with DBAD, Mitsunobu products can be synthesized in typically > 90 % yields without the need for chromatographic purification.

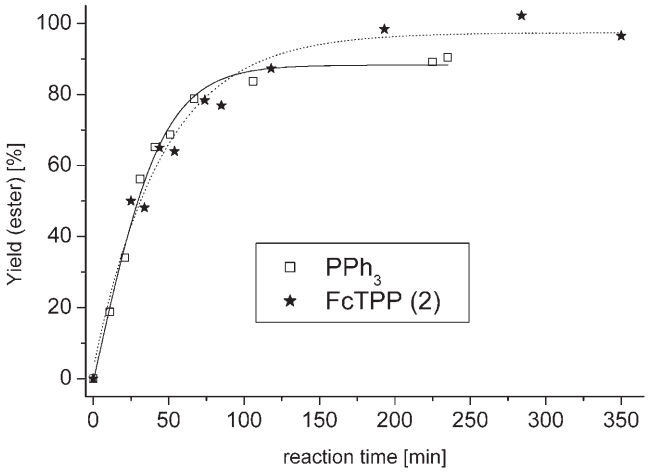


Figure 2. Comparison of reactivity for PPh_3 and the ferrocenyl-tagged PPh_3 (**2**); reaction temperature 0 °C; determination of yield *via* GC using isooctane as an internal standard.

Experimental Section

General Remarks

All chemicals were purchased as reagent grade from commercial suppliers and used without further purification, unless otherwise noted. THF was distilled over potassium and benzophenone under an argon atmosphere, Toluene was distilled over sodium and benzophenone under an argon atmosphere. Proton (^{1}H NMR), carbon (^{13}C NMR) and phosphorus (^{31}P NMR) nuclear magnetic resonance spectra were recorded on a Bruker DRX 500 at 500 MHz, 125.75 MHz and 202.46, respectively, and are referenced to tetramethylsilane ($\delta = 0.0$ ppm), for ^{1}H and ^{13}C NMR, and to H_3PO_4 ($\delta = 0.0$ ppm) for ^{31}P NMR. Thin layer chromatography: Fluka silica gel 60 F 254 (0.2 mm) on aluminum plates. Column chromatography: E. Merck silica gel 60 (0.063–0.20 mesh ASTM). Mass spectra (MS) were recorded on an Agilent 1100 HPLC-MS (column: YMC J′Sphere ODS H80, 4 μm, 20 × 2.1 mm; flow: 1.0 mL min^{-1}; $T = 30$ °C; eluent: (A: water + 0.05 % TFA/B: acetonitrile + 0.05 % TFA) 0.00 min: 4 % B → 2.00 min: 95 % B → 2.45 min: 4 % B); detection: UV + MS (ESI/quadrupole). GC experiments were run on a Perkin–Elmer AutoSystem, CP-Sil (CB, l = 15 m, $d_i = 0.25$ mm, $d_F = 1$ μm), N_2 (flow: 17 cm/sec; split 1:20), FID. Cyclic voltammetry: EG&G 263 A-2 potentiostat. All cyclic voltammograms were recorded in dry CH_2Cl_2 under an argon atmosphere at ambient temperature. A three-electrode configuration was employed. The working electrode was a Pt disk (diameter 1 mm) sealed in soft glass with a Pt wire as counterelectrode. The pseudoreference electrode was an Ag wire. Potentials were calibrated internally against the formal potential of octamethylferrocene

[10 mV (CH_2Cl_2) vs. Ag/AgCl]. NBu_4PF_6 (0.1 mol/L) was used as supporting electrolyte. *p*-Bromophenylferrocene was first prepared by Rosenblum et al.[16]

p-Bromophenylferrocene (1)

In a 1 L round-bottom flask 4-bromoaniline (52.5 g, 339 mmol) was suspended in 300 mL half-concentrated sulfuric acid. Within 30 min. 135 mL of an aqueous solution of $NaNO_2$ (2.5 mol/L) were added at 0 °C. After stirring for an additional 30 min at 0 °C and securing an excess of HNO_2 by use of KI-starch paper, the resulting clear yellow diazonium solution was added to a vigorously stirred solution of ferrocene (21.0 g, 113 mmol) in 600 mL Et_2O and Aliquat 336 (9 g) within 30 min. The reaction mixture was stirred vigorously for 1.5 h at 0 °C and for 30 min at ambient temperature. The reaction mixture was transferred in a separation funnel and the aqueous phase was extracted with Et_2O (3×200 mL). The combined organic phases were washed subsequently with 200 mL water, then 200 mL brine and finally dried with $MgSO_4$. Filtration and concentration under vacuum gave the crude product which was dissolved in 25 mL ethyl acetate and adsorbed on silica gel. Column chromatography (silica, cyclohexane) afforded pure **1** (CAS 58482–65–8) as brown crystals; yield: 28.5 g (74 %); R_f 0.44 (cyclohexane). ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 7.39$ (d, $^3J = 9.0$ Hz, 2H, arom), 7.32 (d, $^3J = 8.5$ Hz, 2H, arom), 4.60 (t, $J = 2.0$ Hz, 2H, Fc), 4.32 (t, $J = 2.0$ Hz, 2H, Fc), 4.03 (s, 5H, Fc); ^{13}C{^{1}H} NMR (125.77 MHz, $CDCl_3$): $\delta = 138.5$, 131.4, 127.6, 119.4, 84.1, 69.7, 69.2, 66.4.

Diphenyl(*p*-ferrocenylphenyl)phosphine (FcTPP) (2)

Grignard route: In a 250 mL round-bottom flask Mg turnings (158 mg, 6.5 mmol) were placed under an argon atmosphere. *p*-Bromophenylferrocene (2.0 g, 5.9 mmol), dissolved in 80 mL dry THF, was added at ambient temperature and sonicated for 12 h. The Grignard solution was transferred into a 250 mL Schlenk-flask, containing Ph_2PCl (1.24 mL, 6.9 mmol), dissolved in dry THF (20 mL). The resulting reaction mixture was stirred for 4 h at ambient temperature. Silica gel (5 g) was added and volatiles were removed under vacuum. Purification *via* column chromatography (silica gel, 100:2 cyclohexane/EtOAc) afforded **2** as a red viscous oil; yield: 2.1 g (81 %); R_f 0.60 (100:2 cyclohexane/EtOAc).

t-BuLi route: In a 100 mL Schlenk flask *p*-bromophenylferrocene (3.0 g, 8.8 mmol) was dissolved in 50 mL absolute THF. Under an argon-atmosphere *t*-BuLi, 1.5 M in pentane (6.0 mL, 8.9 mmol) was added at −78 °C within 10 min. The now brownish solution was stirred for additional 2 h at that temperature before $PhPCl_2$ (1.75 mL, 2.15 g, 9.75 mmol) was added. The reaction mixture was stirred for 1 h at −78 °C and further 2 h at ambient temperature. Silica gel (5 g) was added and volatiles were removed under vacuum. Purification *via* column chromatography (silica gel, 100:2 cyclohexane/EtOAc) afforded **2** as a red viscous oil; yield: 3.25 g (83 %); R_f 0.60 (100:2 cyclohexane/EtOAc). ^{1}H NMR (acetone-d_6): $\delta = 7.43$–7.07 (m, 14H, arom), 4.62 (t, $J = 1.8$ Hz, 2H, Fc), 4.21 (t, $J = 1.8$, 2H, Fc), 3.88 (s, 5H, Fc); ^{13}C{^{1}H} NMR (acetone-d_6): $\delta = 141.9$, 139.0 (d, $J_{P,C} = 12.3$ Hz), 138.9 (d, $J_{P,C} = 9.9$ Hz), 134.9, 134.7, 130.1, 129.8 (d, $J_{P,C} = 11.9$ Hz), 127.4 (d, $J_{P,C} = 6.8$ Hz), 85.6, 70.8, 70.5, 67.8; ^{31}P NMR (acetone-d_6): $\delta = -7.81$; anal. calcd. for $C_{28}H_{23}FeP$ (446.3): C 75.4, H 5.20; found: C 75.3, H 4.95.

General Mitsunobu Procedure

To a stirred solution of the nucleophile (carbonic acid or phenol or phthalimide) (1.3 mmol), FcTPP **2** (1.3 mmol) and the alcohol (1.0 mmol) in dry THF (3 mL), di-*tert*-butyl azodicarboxylate (1.3 mmol) dissolved in dry THF (2 mL) was added. The resulting solution was stirred overnight at ambient temperature. The reaction mixture was treated with $FeCl_3$ (500 mg) and stirred for 5 min, then Et_2O (10 mL) and water (10 mL) were added. The separated organic phase was treated again with $FeCl_3$ (200 mg), stirred for 5 min and washed with water. Then 5 mL HCl (4.0 mol/L in dioxane, 20 mmol) were added to the slightly yellow organic phase and stirred for 1 h. The solution was washed with water (2×20 mL), dried with $MgSO_4$ and filtered. Volatiles were removed under vacuum. The crude product was filtered through a pad of silica gel (5 cm, eluent: cyclohexane/ethyl acetate, 10:1). Volatile compounds of the filtrate were removed under vacuum, affording the respective products as slightly yellow oils (> 90 % isolated yield).

Cyclopentyl 2-chlorobenzoate (CAS 501356–76–9): ^{1}H NMR ($CDCl_3$): $\delta = 7.77$ (dd, $^3J = 5$ Hz, $^4J = 1.5$ Hz, 1H, *o*-C*H*, arom), 7.44–7.37 (m, 3H, C*H*, arom), 5.44 (m, 1H, COOC*H*), 1.97–1.64 (m, 8H, CH_2); ^{13}C{^{1}H} NMR (125.77 MHz, $CDCl_3$): $\delta = 165.7$, 133.4, 132.2, 131.2, 131.0, 130.9, 126.5, 78.6, 32.8, 23.8.

***n*-Butyl 3-*tert*-butylphenyl ether** (CAS 136–60–7): ^{1}H NMR ($CDCl_3$): $\delta = 7.14$ (t, $^3J = 8$ Hz, 1H, *m*-C*H*, arom), 6.90–6.86 (m, 2H, C*H*, arom), 6.65–6.62 (m, 1H, C*H*, arom), 3.89 (t, $^3J = 6.5$ Hz, 2H, O-CH_2), 1.73–1.67 (m, 2H, CH_2), 1.47–1.39 (m, 2H, CH_2), 1.24 (s, 9H, *t*-Bu), 0.91 (t, $^3J = 8$ Hz, 3H, CH_2CH_3); ^{13}C{^{1}H} NMR ($CDCl_3$): $\delta = 158.0$, 151.9, 127.9, 116.6, 111.6, 109.5, 66.5, 33.7, 30.5, 30.3, 18.3, 12.9; MS: $m/z = 207$ $[M+H]^+$.

3-Prop-2-ynyl 3-*tert*-butylphenyl ether: ^{1}H NMR ($CDCl_3$): $\delta = 7.16$ (t, $^3J = 7.5$ Hz, 1H, *m*-C*H,* arom), 6.96–6.94 (m, 2H, C*H,* arom), 6.73–6.70 (m, 1H, C*H,* arom), 4.61 (d, $^4J = 2.5$ Hz, 2H, O-CH_2), 2.43 (t, $^4J = 2.5$ Hz, 1H, C≡C*H*), 1.23 (s, 9H, *t*-Bu); ^{13}C{^{1}H} NMR ($CDCl_3$): $\delta =$ 156.4, 152.1, 127.9, 117.7, 112.0, 109.9, 77.8, 74.3, 54.7, 33.7, 30.2; MS: $m/z = 189$ $[M+H]^+$.

***sec*-Hexyl 3-*tert*-butylphenyl ether:** ^{1}H NMR ($CDCl_3$): $\delta = 7.12$ (t, $^3J = 7.5$ Hz, 1H, *m*-C*H*, arom), 6.89–6.84 (m, 2H, C*H*, arom), 6.63–6.61 (m, 1H, C*H*, arom), 4.27 (tq, $^3J =$ 6 Hz, 1H, O-C*H*), 1.71–1.65 (m, 1H, O-CHCH_2), 1.52–1.46 (m, 1H, O-CHCH_2), 1.41–1.29 (m, 4H, CH_2), 1.23 (s, 9H, *t*-Bu), 1.22 (d, $^3J = 7$ Hz, 3H, O-CHCH_3), 0.84 (t, $^3J = 7.5$ Hz, 3H, CH_2CH_3); ^{13}C{^{1}H} NMR ($CDCl_3$): $\delta = 157.0$, 151.9, 127.8, 116.5, 113.0, 110.9, 72.6, 35.3, 33.7, 30.3, 26.8, 21.7, 18.8, 13.0; MS: $m/z = 235$ $[M+H]^+$.

4-Bromobenzyl chloroacetate: ^{1}H NMR ($CDCl_3$): $\delta = 7.42$ (d, $^3J = 8.5$ Hz, 2H, CH, arom), 7.16 (d, $^3J = 8.5$ Hz, 2H, CH, arom), 5.08 (s, 2H, O-CH_2), 4.01 (s, 2H, $COCH_2$); ^{13}C{^{1}H}

NMR ($CDCl_3$): $\delta = 166.0$, 132.9, 130.8, 129.1, 121.8, 66.0, 39.8; MS: $m/z = 212$, 214 $[M\text{-}CH_3Cl]^+$.

***n*-Propyl *p-sec*-butoxybenzoate:** ^1H NMR ($CDCl_3$): $\delta =$ 7.98 (d, $^3J = 9.3$ Hz, 2H, C*H,* arom), 6.89 (d, $^3J = 9.0$ Hz, 2H, C*H,* arom), 4.38 (tq, $^3J = 6$ Hz, 1H, O-C*H*), 4.24 (t, $^3J =$ 6.3 Hz, 2H, $COOCH_2$), 1.83–1.30 (m, 4H, CH_2), 1.31 (d, $^3J = 6$ Hz, 3H, $CHCH_3$), 1.04–0.94 (m, 6H, CH_2CH_3); $^{13}C\{^1H\}$ NMR ($CDCl_3$): $\delta = 166.5$, 162.1, 131.6, 122.5, 115.0, 75.1, 66.2, 29.1, 22.2, 19.1, 10.6, 9.7; MS: $m/z = 237$ $[M+H]^+$.

***N*-*sec*-Hexylphthalimide** (CAS 221155–51–7): ^1H NMR ($CDCl_3$): $\delta = 7.74$–7.72 (m, 2H, C*H*, arom), 7.63–7.61 (m, 2H, C*H*, arom), 4.26 (tq, $^3J = 7$ Hz, 1H, N-C*H*), 2.02–1.94 (m, 1H, $CHCH_2$), 1.69–1.62 (m, 1H, $CHCH_2$), 1.38 (d, $^3J =$ 7 Hz, 3H, $CHCH_3$), 1.29–1.07 (m, 4H, CH_2), 0.77 (t, $^3J =$ 7.5 Hz, 3H, CH_2CH_3); $^{13}C\{^1H\}$ NMR ($CDCl_3$): $\delta = 167.5$, 132.7, 131.0, 122.0, 46.5, 32.4, 27.9, 21.3, 17.7, 12.9; MS: $m/z = 233$ $[M+H]^+$.

***N*-2-Ethylhexylphthalimide:** ^1H NMR ($CDCl_3$): $\delta = 7.85$–7.82 (m, 2H, C*H*, arom), 7.72–7.70 (m, 2H, C*H*, arom), 3.58 (d, $^3J = 7.5$ Hz, 2H, N-CH_2), 1.84 (tt, $^3J = 6.5$ Hz, 1H, N-CH_2C*H*), 1.41–1.25 (m, 8H, CH_2), 0.92 (t, $^3J = 7.5$ Hz, 3H, ethyl-CH_3), 0.88 (t, $^3J = 7.5$ Hz, 3H, hexyl-CH_3); $^{13}C\{^1H\}$ NMR ($CDCl_3$): $\delta = 167.7$, 132.8, 131.1, 122.1, 40.9, 37.3, 29.5, 27.5, 22.9, 22.0, 13.0, 9.4; MS: $m/z = 260$ $[M+H]^+$.

Recovery of FcTPPO (3)

The combined aqueous phases of a Mitsunobu reaction (as described above), containing Diphenyl-FcTPPO$^+$ (**3**$^+$) were treated with saturated $Na_2S_2O_3$ solution (50 mL). After addition of THF (30 mL) the mixture was stirred for 15 min at ambient temperature, then extracted with Et_2O (3×30 mL). The combined organic phases were washed with brine, dried with $MgSO_4$ and filtered. Removal of the volatiles under vacuum afforded 580 mg of crude product. Further purification through a short plug of silica gel (6 cm, elution of impurities with cyclohexane/ethyl acetate, 10:1, then elution of the ferrocene with cyclohexane/ethyl acetate, 1:9) afforded pure FcTPPO as a red-orange solid; yield: 500 mg (1.08 mmol, 83%); R_f 0.38. (1:9 cyclohexane/EtOAc). ^1H NMR (benzene-d_6): $\delta = 7.86$–7.82 (m, 4H, arom), 7.76–7.72 (m, 2H, arom), 7.30–7.27 (m, 2H, arom), 7.08–7.02 (m, 6H, arom), 4.41 (t, $J = 2.0$ Hz, 2H, Fc), 4.11 (t, $J = 2.0$, 2H, Fc), 3,82 (s, 5H, Fc); $^{13}C\{^1H\}$ NMR (DMSO-d_6): $\delta = 144.1$, 133.8, 133.0, 132.3, 132.0, 131.84 (d, $J_{P,C} = 9.3$ Hz), 129.1 (d, $J_{C,B} = 11.1$ Hz), 126.3 (d, $J_{P,C} = 12.3$ Hz), 83.3, 70.0, 69.9, 67.2; ^{31}P NMR (acetone-d_6): $\delta = 25.70$; ^{31}P NMR (DMSO-d_6): $\delta = 23.99$; anal. calcd. for $C_{28}H_{23}FeOP$ (462.3): C 72.8, H 5.02; found: C 73.9, H 5.55.

Reduction of FcTPPO (3)

FcTPPO (**3**) (450 mg, 0.97 mmol) was dissolved in absolute toluene (7 mL) and placed in a pressure tube under an argon atmosphere. TEA (1.5 mL) and $HSiCl_3$ (1.0 mL) were added and the reaction mixture was stirred at 120 °C for 12 h. The mixture was cooled to room temperature, 20 mL H_2O were added before extraction with Et_2O (3×20 mL). The combined organic phases were washed subsequently with water (15 mL) and brine (15 mL), dried with $MgSO_4$ and filtered. Removal of volatiles under vacuum afforded **2** as a red viscous oil; yield: 431 mg (100%). ^1H, ^{13}C and ^{31}P NMR spectra were found to be identical with those described above for FcTPP.

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